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Interim Technical Report No. 3

RATE OF DIFFUSION OF CARBON IN ALPHA AND IN BETA TITANIUM AS A FUNCTION OF THE TEMPERATURE AND CONCENTRATION

Contract No. DA-36-034-ORD-1157 (RD)

Submitted to: Watertown Arsenal Laboratory
Ordnance Department
Watertown 72, Mass.

Submitted by: Horizons Incorporated
Cleveland 4, Ohio

Date: January 18, 1954

Interim Technical Report No. 3 1955
(Period June 12, 1953 - December 31, ~~1954~~)

RATE OF DIFFUSION OF CARBON IN ALPHA AND IN BETA TITANIUM AS A FUNCTION OF THE TEMPERATURE AND CONCENTRATION

For

Watertown Arsenal
Watertown, Massachusetts
Contract DA-36-034-ORD-1157 (RD)

By

Horizons Incorporated
Cleveland, Ohio

Date Report Completed: January 18, 1954

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(4) Number of Report on Project: ORDTB 2-1065

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(6) Title of Project: RATE OF DIFFUSION OF CARBON IN ALPHA AND IN BETA TITANIUM AS A FUNCTION OF THE TEMPERATURE AND CONCENTRATION

Contract Number: DA-36-034-ORD-1157 (RD)

(7) Object:

To determine the diffusion rate of carbon in titanium, in both the alpha and beta regions.

(8) Summary:

The experimental program on the determination of the rate of diffusion of carbon in titanium has progressed to the point where an accurate analysis of the data can be accomplished. The diffusion of carbon in titanium in both the alpha and beta regions was determined by the Van Orstrand-Dewey type of treatment. It is believed that the concentration curve follows the equation:

SUMMARY (continued)

$$C = C_{1.0} - B\left(1 + \operatorname{erf}\frac{x}{2\sqrt{Dt}}\right)$$

when x is less than the interface movement (ϵ).

For two specimens annealed at the same temperature of 736° C, the same diffusion coefficients were obtained from an analysis of the data. This tentatively establishes that the ϵ is directly proportional to the square root of the time at temperature.

A logarithmic plot of the diffusion coefficient versus the reciprocal of the absolute temperature, for the data obtained, results with two parallel straight lines for the alpha and beta regions. The experimental activation energy in the alpha and beta range was therefore found to be the same. (44,600 cal. mole^{-1}) The frequency factors were found to be:

$D_0 = 8.63$ in the α range

$D_0 = 27.75$ in the β range.

By substituting the D_0 values for the alpha and beta temperatures in the Arrhenius type equation,

$$D = D_0 e^{-Q/RT}$$

the diffusion coefficients may be calculated. For example:

For α temperature of 835° C,

$$D = 8.63\, e^{-\frac{44,600}{1.99 \times 1107}}$$

$$D = 1.34 \times 10^{-8}\ \text{cm}^2\ \text{sec}^{-1}$$

SUMMARY (continued)

For β temperature of 950° C,

$$D = 27.75\, e^{-\frac{44,600}{1.99 \times 1222}}$$

$$D = 3.07 \times 10^{-7}\ cm^2\ sec^{-1}$$

The final analysis of the completed results will determine the validity of the presented results shown in Table I.

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I. INTRODUCTION

The text of this report deals with the experimental program for determining the rates of diffusion of carbon in titanium for both the alpha and beta regions.

Knowledge of the diffusivity of carbon in titanium is useful in the thermal treatment of titanium carbon alloys.

Briefly, one of the applications would be for accurate determination of alpha and beta solubility limits for their respective temperature range. The stability of the alpha and beta phase may also be determined. In some cases where an inhomogeneous casting contains concentrated phases of carbon, a specific time at temperature can be determined for homogenization.

In heat treating where high purity carbon is packed around titanium and annealed under an inert atmosphere or vacuum, the case depth or concentration gradient of the case can be predetermined for a given temperature and time. With this information the mechanical and physical properties may be specified and met, thereby enhancing the many applications for which titanium would be ideally suited.

The presented results are few admittedly; however, they are in agreement with each other and show some indication of what may be expected. This experimental program is in the final stage and the remaining experiments will

determine the validity of the presented results.

II. LITERATURE

Literature on the treatment of the diffusion equations is rather extensive; however, very little information is available on the treatment of diffusion problems in binary systems consisting of more than one phase. There is no existing data, in particular, on the diffusion of carbon in alpha and beta titanium.

A. Theory

The diffusion studies are made with a diffusion couple consisting of (a) high purity titanium .0383% C, and (b) 1.300% C alloy titanium. Thus, at most temperatures, excepting those between the allotropic transformation temperature (882° C) and the peritectoid temperature (920° C), the diffusion will occur from a conglomerate of $\alpha + \delta$ into α or from a conglomerate of $\beta + \delta$ into β as shown in Figure 1. The mechanism of diffusion in both of these cases is the same and can be treated in the same manner. The Van Orstrand-Dewey[1] method consists of allowing carbon to diffuse from an unexhaustible supply into an essentially carbon-free specimen (see Figure 2). W. Jost[2] and C. Wagner[3] treat this method in a manner which is practicable. If a diffusion couple composed of pure titanium with a .038 per cent carbon content and titanium alloy with a 1.300 per cent carbon content is vacuum annealed at 800° C, carbon will diffuse out of the $\alpha + \delta$ into the α region. This is slowly depleting the δ region of carbon

and results in a shift of the plane of discontinuity as shown in Figure 3. The diffusion will proceed in the α phase only, and obey Fick's second law:

$$\frac{\partial c}{\partial t} = D_{\alpha} \frac{\partial^2 c}{\partial x^2} \qquad x < \epsilon \qquad (1)$$

Provided that D is independent of concentration.

A particular integral of this equation is,

$$C = C_{1,0} - B\left(1 + \operatorname{erf} \frac{x}{2\sqrt{Dt}}\right) \qquad (2)$$

for $x < 0$

the initial conditions being,

$C = C_{1,0}$ at $x < 0$ and $t = 0$

$C = C_0$ at $x > 0$ and $t = 0$

the mass flow equation for this case is,

$$(C_0 - C_{1,2})\, d\epsilon = -D_{\alpha}\, dt \left(\frac{\partial c}{\partial x}\right) \qquad (3)$$

the remaining boundary conditions are,

$C = C_{1,2}$ at $x = \epsilon_{-0}$ and $t > 0$

$C = C_0$ at $x = \epsilon_{+0}$ and $t > 0$

assuming the relationship,

$$\epsilon = 2\gamma\sqrt{Dt} \qquad (4)$$

and combining equations (2) with (3) and (4) along with the boundary conditions, the resulting equation is,

$$\frac{C_{1,0} - C_{1,2}}{C_{1,2} - C_0} = \sqrt{\pi}\,\gamma e^{\gamma^2}\left(1 + \operatorname{erf}\gamma\right) \qquad (5)$$

This solution was used for diffusion in both the α and β temperature

range. The entire derivation assumed D independency of concentration; however, the maximum solubility of carbon in titanium is only about 0.38 per cent and, therefore, the dependence of the diffusion coefficient on concentration should be very small.

B. Activation Energy Determination

Determination of the activation energy was done by assuming that D varies with temperature in accordance with the Arrhenius type equation,

$$D = D_0 e^{-\frac{Q}{RT}} \qquad (6)$$

where D_0 is the frequency factor and varies very little with temperature and,

Q is the experimental activation energy,

R is the gas constant, and

T is the absolute temperature.

By taking the slope of a logarithmic plot of D versus 1/T and equating this to Q/R and knowing D and Q, D_0 can be calculated from this equation.

III. EXPERIMENTAL PROCEDURE

The procedure for the preparation of a specimen's surface for butt welding was covered in our Interim Technical Report No. 2. A review of the procedure is included for the purpose of stressing the care involved in preparations.

A. Butt Welding Apparatus

A 440 V butt welder was used for the fusion welding of the specimens which are .750 inch diameter by 2 inches in length each. The complete procedure for preparation and welding of specimens was as follows:

1. Polishing of contact surfaces through 3/0 metallographic paper.
2. Cleaning of contact surfaces by etching with a 2% hydrofluoric - 10% nitric acid solution.
3. Successive flushing of welding chamber (see Figure 4) with argon and leaving a positive argon pressure in chamber during welding operation.
4. Applying an initial load of 3000 lbs. to the couple for the purpose of insuring good contact.
5. Power turned on at a setting of 13,830 amps per square inch and a voltage drop of 2.82 across the specimen.
6. Power turned off after proper flow at the interface - this is done by watching the load drop to 1500 lbs.

The welding time is approximately 8 seconds. The fusion along the interface must be complete with no oxidation to permit ideal diffusion during the annealing operation.

B. Vacuum Diffusion Furnaces

The vacuum furnaces are of the vertical tube type as shown in Figure 5. Globar resistance heating elements are used for temperatures

above 950° C, while nichrome resistance wound elements are used for the α temperature range studies. By controlling both type furnaces at the heat source and by paralleling an additional resistance across the power relays, temperature fluctuations within the heating chambers themselves was held within $\pm$ 2-1/2° F. The thermocouples inside the heating chambers are connected to an automatic Brown recorder for maintaining a permanent record.

C. Obtainable Vacuums

The vacuums obtained and held during annealing operations has been 1×10^{-4} mm pressure of Hg or less. At room temperature the vacuums of all four furnaces were 2×10^{-5} mm pressure of Hg or less.

D. Sectioning of Specimen

After annealing the specimens they were polished across the interface and prepared for metallographic inspection. With the use of a filar eyepiece the movement of the interface was very accurately determined. (see Figures 6, 7, 8, 9) After the determination of the interface movement the specimens were machined in layers for carbon analysis as shown in Figure 10. The layers are machined on a lathe and after each slice visual inspection on a comparator determined the thickness of each layer, individually and accumulatively, with an accuracy of $\pm$ .0002 inch. The chips for each layer are collected on aluminum foil to prevent any carbon contamination. The lathe is thoroughly cleaned before sectioning is started for the same purpose. Each

layer contains enough material for two one gram specimens for carbon analysis.

E. Material Analysis

The material used for the couples was iodide titanium and iodide titanium alloyed with carbon. The chemical analysis of the iodide material was:

C = .037%
N = .006%
Fe = .013%

The oxygen analysis has not been received to date. The 1.300 per cent carbon alloy was made by adding carbon to half of the iodide titanium. The chemical analysis of this alloy would therefore be:

C = 1.300%
N = .006%
Fe = .013%

Some oxygen and hydrogen was probably picked up during the alloying operation. The complete spectrochemical analyses of these materials will be given in the next report.

All carbon determinations were run on a semi-automatic carbon determinator which is manufactured by the Laboratory Equipment Corporation. The principle of the apparatus is, namely, the combustion of a given metal sample followed by the collection of the evolved gases, adsorption of the CO_2, and measurement of this loss in volume. The obtainable accuracy is $\pm$ .005 per cent carbon.

F. Errors

Sources of error are practically unavoidable in the conducting of diffusion experiments. By estimating the welding temperature at the interface to be approximately 900° C and the time at temperature to be 2 seconds, it can be seen that the diffusion occurring during welding is negligible since the order of the diffusion rate is 1×10^{-7} cm^2 sec^{-1} at this temperature. Some diffusion is also caused by the heating and cooling cycle for each diffusion anneal. However, since the diffusion rate is more than ten times greater for each 100° C in the annealing range and since the time for heating and cooling of the specimen is very small (about 2 hours) as compared to the actual time at temperature, this is not serious. Measurement of the interface movement is done by metallographic examination with an accuracy of $\pm$ .002 mm. The temperature control is $\pm$ 2-1/2° F, which averages out fairly well since the zero point is set on temperature. Measurement and successive machining of the layers for carbon analysis also introduces an error of $\pm$ .005 per cent carbon. This is averaged out to some degree when the curve is drawn through the experimental points. This source of errors does not influence the diffusion coefficient determination if the solubility limit is known for the annealing temperature.

The accumulation of all these errors is still fairly small. Any attempt to apply a correction factor is therefore deemed impracticable in the presented program.

IV. RESULTS

The diffusion anneals for specimens in the alpha region will be conducted at 736° C, 782° C, and 835° C; for specimens in the beta region 950° C, 1050° C, and 1150° C. Fifteen couples have been annealed and six of these have been analyzed for D values at the following times and temperatures:

Spec. No.	Temp., °C	Time (hours)
1	835	100
2	782	400
3	736	595
4	1050	140
5	950	140
6	736	1006

Figures 6, 7, 8, and 9 distinctly show the observed interface shift. (Also see Table II) The weld is shown at the bottom of the photomicrographs with the α or β regions being clearly bounded by the δ phase. By knowing this interface movement (ϵ), by metallographic measurement, the specimen can be machined in sections for carbon analysis. The carbon analysis for the first three specimens are shown in Tables III, IV, and V. The carbon analysis on the remaining specimens are being completed at the present time. It can be seen by the small interface movement (ϵ) that the high carbon content of 1.300 per cent makes it difficult for accurate determinations of the α solubility limit (see Figure 11). For this reason values for the α solubility limit ($C_{1,2}$) at the respective temperatures were taken from the WADC Technical Report 53-41. (see Figure 1) It is believed that a fairly

accurate α solubility limit will be obtained from a specimen annealed at 835° C for 1000 hours. When the specimens annealed in the β region have been analyzed, a fairly accurate β solubility limit will be determined for 950° C, 1050° C, and 1150° C. For the purpose of calculating the diffusion coefficient D, however, these solubility limits were approximated from the above mentioned report.

For specimens 1, 2, 3, and 6 the values of the parameter γ were determined by substituting in equation (5):

$$\frac{C_{1,0} - C_{1,a}}{C_{1,2} - C_0} = \sqrt{\pi}\,\gamma\, e^{\gamma^2} (1 + \operatorname{erf}\gamma)$$

then by substituting the calculated γ into equation (4):

$$\epsilon = 2\gamma\sqrt{Dt}$$

the diffusion coefficients were determined for the α range. The same was done for the β range specimens. (See Table VI) By knowing D, the carbon contents, and the α solubility limit at 835° C, the concentration curve for specimen No. 1 was drawn according to equation (2):

$$C = C_{1,0} - B\left(1 + \operatorname{erf}\frac{x}{2\sqrt{Dt}}\right)$$

The experimental points were in turn fixed on the curve according to their relative positions to check the validity of the theory. It can be seen from Figure 11 that a larger interface shift is needed to adequately determine the experimental curve.

Specimens 3 and 6 were annealed at the same temperature 736° C for 595

and 1006 hours, respectively, and have the same value for the diffusion coefficient. This gives some justification to the assumption that:

$$\epsilon = 2\delta\sqrt{Dt}$$

However, more results are needed to validify the relationship.

The activation energy for the α and β regions was calculated from the slope of the curve D vs. 1/T as shown in Figure 12. The slope of the line was the same in the α and β range; however, here again only two experimental points have been determined in the β region and more data is needed to justify the line drawn. The experimental activation energy was found to be 44,600 cal per mole. By knowing the D, T, and Q values, the frequency factor was determined for the alpha and beta regions to be 8.63 and 27.75 respectively.

V. FUTURE WORK

The remaining time of this experimental program on the diffusion rates of carbon in titanium in both the alpha and beta regions will be used for:

1. Heat treatment of the remaining diffusion couples.
2. Heat treatment of a few 1/2 inch diameter couples of high purity titanium and .49 per cent carbon titanium alloy for verification of D independency of concentration.
3. Machining and carbon analysis of the specimens.
4. Data analysis.
5. Presentation of results in the final report.

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2	Jost, W., Diffusion in Solids, Liquids, Gases, 1952, Academic Press Inc., Publishers, New York, p. 68-72.
3	Wagner, C., Unpublished results. W. Jost reproduced the calculations in above book, p. 69.

TABLE I

Spec. No.	Temp. °C	Time (hrs.)	D	% C_{111}	ϵ cm
1	835	100	1.34×10^{-8}	.316	.0182
2	782	400	5.53×10^{-9}	.258	.0188
3	736	595	1.98×10^{-9}	.240	.0125
4	1050	140	1.25×10^{-6}	.160	.0899
5	950	140	3.07×10^{-7}	.160	.0446
6	736	1006	1.98×10^{-9}	.240	.0163

Range	Q cal/mole	D_o
α	44,600	8.63
β	44.600	27.75

TABLE II

Specimen No.	Interface Movement ϵ mm
1	.182
2	.188
3	.125
4	.899
5	.446
6	.163

TABLE III

Specimen No. 1

Slice No.	%C	Layer (in.)	Distance from end to center of slice (cm)
1-1	.042	.0775	.0984
1-2	.038	.0790	.2970
1-3	.037	.0780	.4960
1-4	.033	.0765	.6930
1-5	.033	.0790	.8900
1-6	.046	.0795	1.0910
1-7	.037	.0765	1.2900
1-8	.042	.0775	1.4850
1-9	.033	.0785	1.6820
1-10	.037	.0775	1.8810
1-11	.042	.0785	2.080
1-12	.051	.0780	2.280
1-13	.246	.0785	2.480
1-14	1.300	.0775	2.675
1-15	1.270	.0775	2.875
1-16	1.320	.0785	3.065
1-17	1.330	.0780	3.265
1-18	1.273	.0780	3.470

TABLE IV

Specimen No. 2

Slice No.	%C	Layer (in.)	Distance from end to center of slice (cm)
2-1	.038	.0780	.0990
2-2	.034	.0775	.2960
2-3	.045	.0785	.4950
2-4	.041	.0775	.6930
2-5	.039	.0775	.8900
2-6	.037	.0780	1.0880
2-7	.037	.0770	1.2850
2-8	.038	.0790	1.4820
2-9	.040	.0780	1.6840
2-10	.040	.0780	1.883
2-11	.037	.0775	2.080
2-12	.037	.0780	2.279
2-13	.311	.0775	2.475
2-14	1.300	.0780	2.670
2-15	1.320	.0775	2.870
2-16	1.290	.0780	3.060
2-17	1.290	.0780	3.265
2-18	1.300	.0780	3.465

TABLE V

Specimen No. 3

Slice No.	%C	Layer (in.)	Distance from end to center of slice (cm)
3-1	.038	.0780	.0991
3-2	.038	.0780	.2974
3-3	.038	.0780	.4960
3-4	.037	.0780	.6940
3-5	.037	.0780	.8915
3-6	.039	.0790	1.0920
3-7	.039	.0770	1.2900
3-8	.038	.0790	1.4900
3-9	.041	.0770	1.6860
3-10	.041	.0775	1.8820
3-11	.038	.0775	2.080
3-12	.040	.0780	2.278
3-13	.196	.0780	2.477
3-14	1.320	.0780	2.672
3-15	1.320	.0770	2.863
3-16	1.290	.0790	3.066
3-17	1.280	.0790	3.266
3-18	1.290	.0770	3.466

TABLE VI

Spec. No.	Temp. °C of anneal	Diff. Coefficient D
1	835	1.34×10^{-8}
2	782	5.53×10^{-9}
3	736	1.98×10^{-9}
4	1050	1.25×10^{-6}
5	950	3.07×10^{-7}
6	736	1.98×10^{-9}

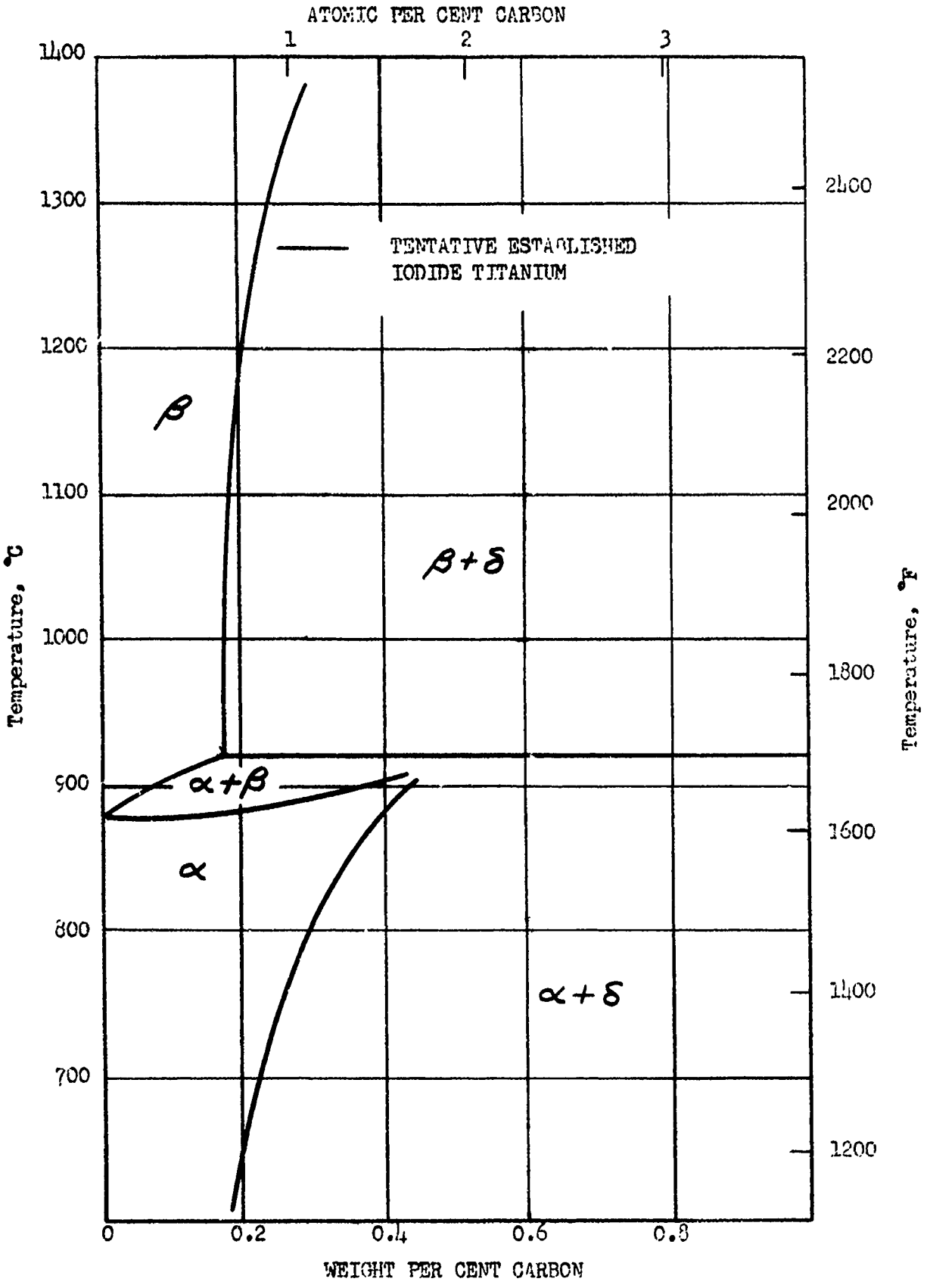


Figure 1. Phase Diagram of Titanium-Carbon System

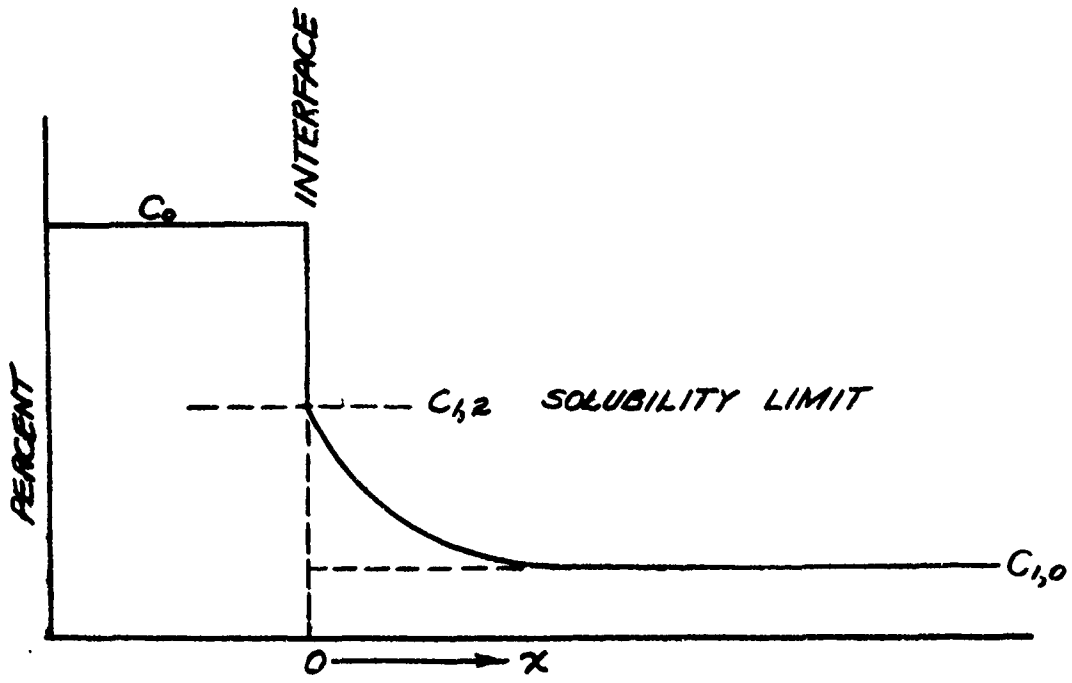


Figure 2. Van Orstrand and Dewey Method

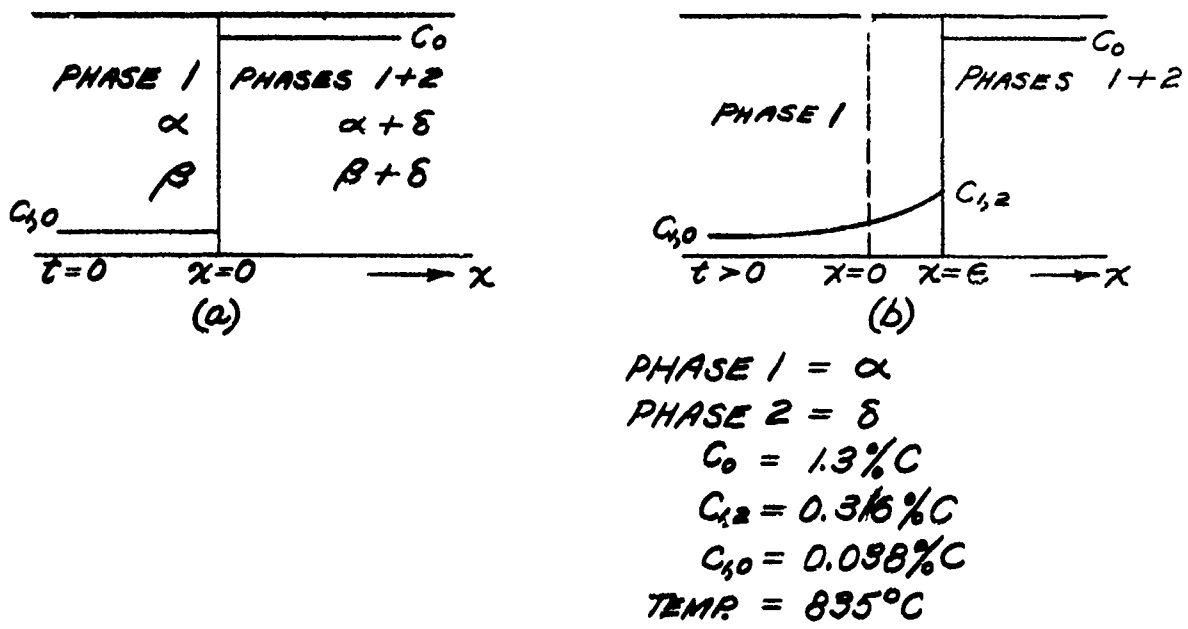


Figure 3. Diffusion in Titanium-Titanium Carbon Couple

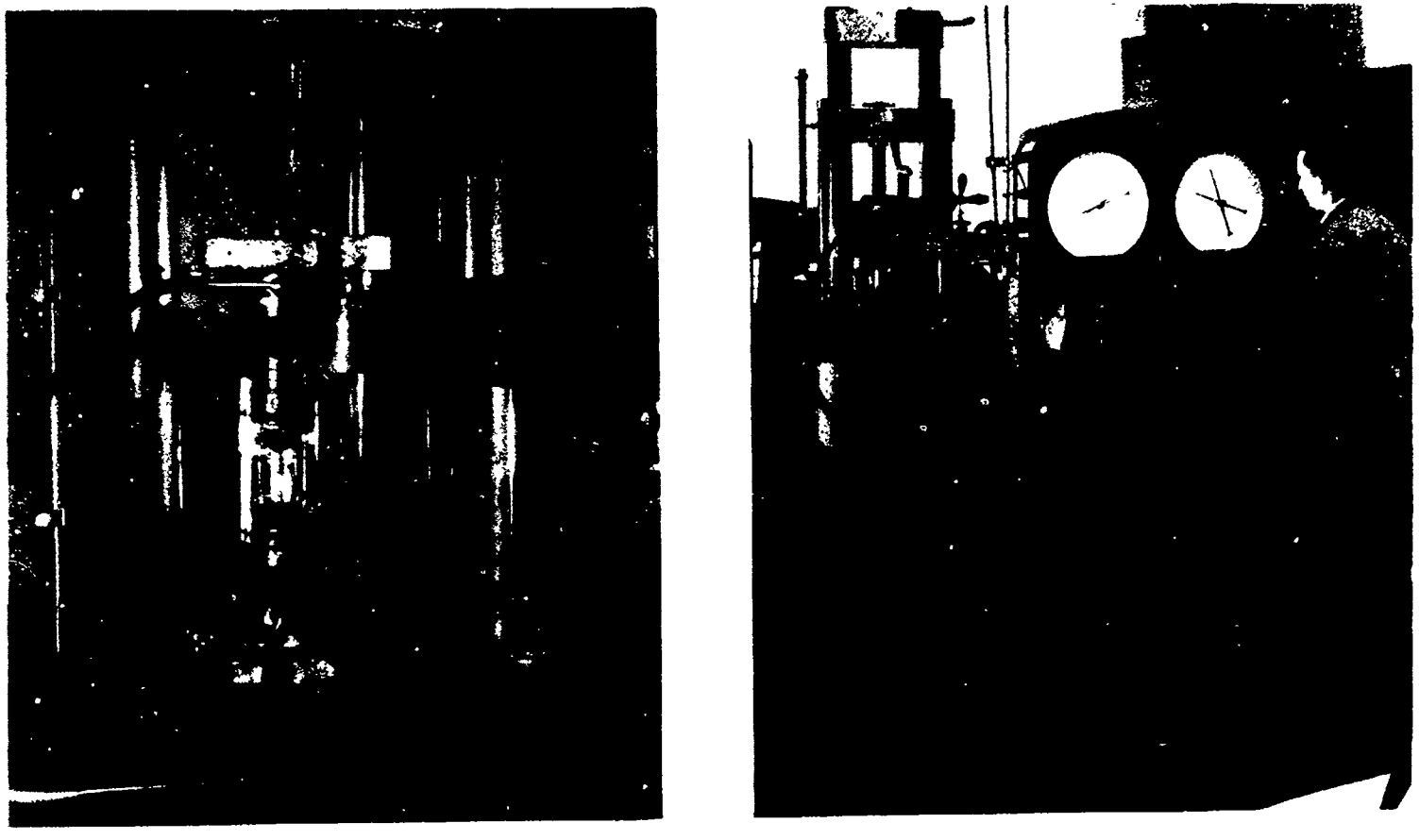


Figure 4. Butt Welding Apparatus

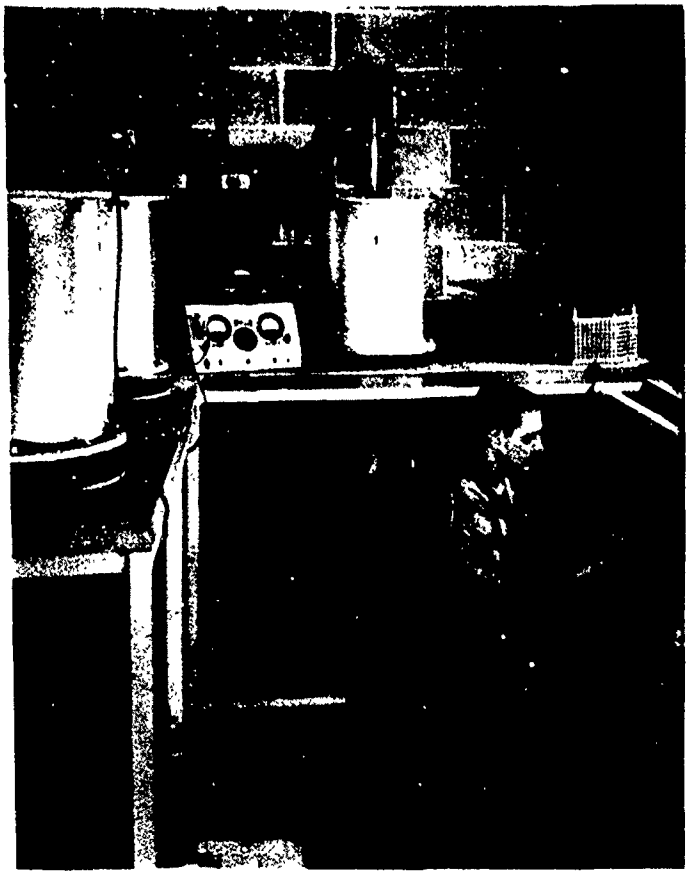


Figure 5. Vacuum Furnaces

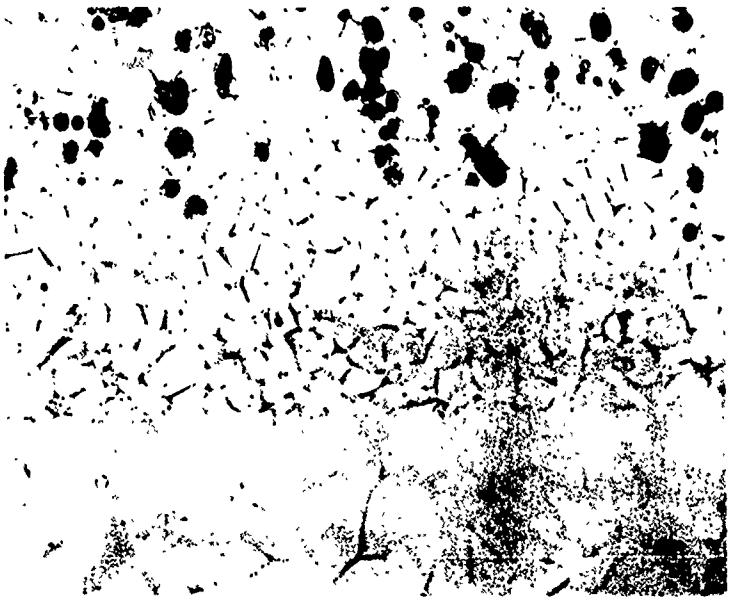


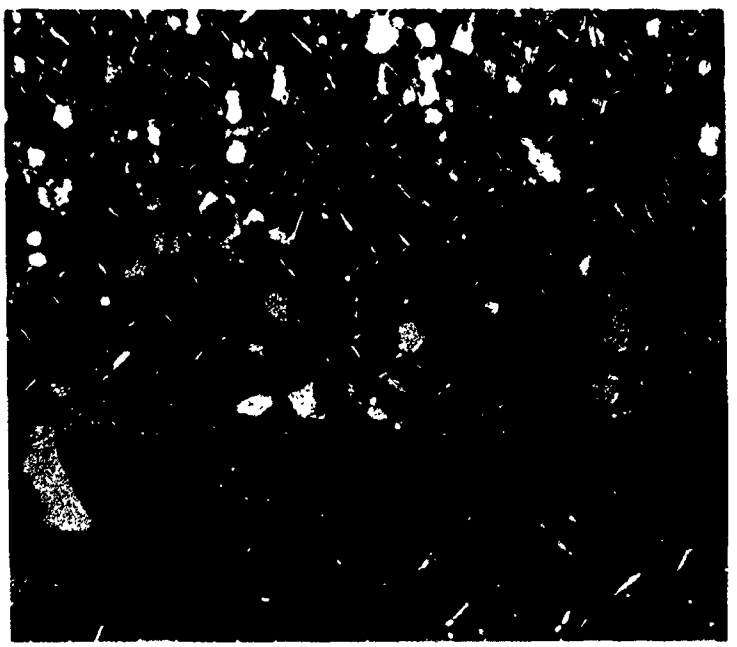
Figure 6

Bright Light

ϵ = .132 mm.

Spec. 1

← Weld



Polarized Light

Spec. 1

835° C - 100 hours

← Weld

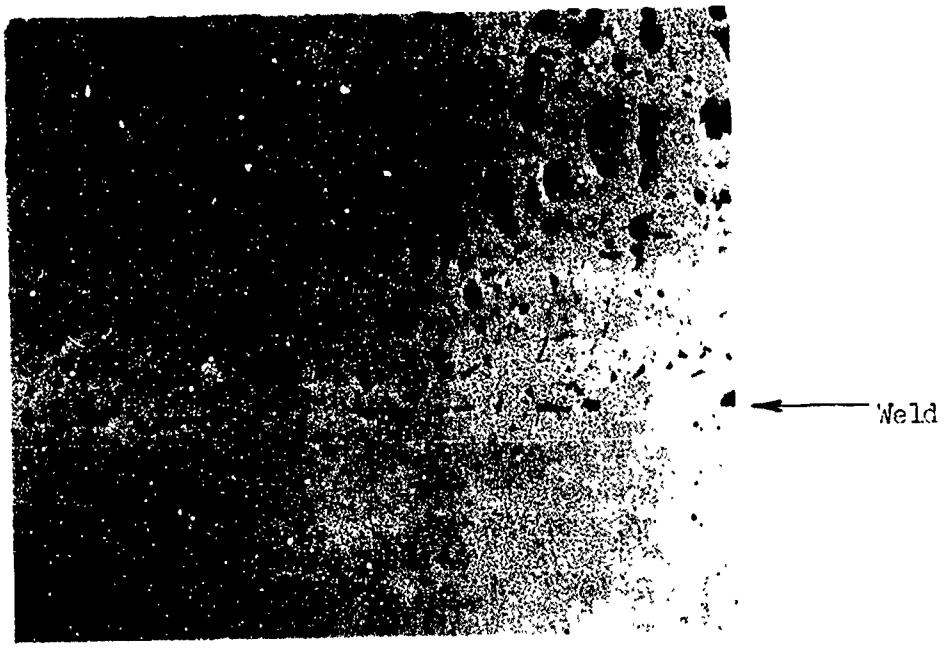


Figure 7

ϵ = .188 mm.

Spec. No. 2 782° C for 400 hours

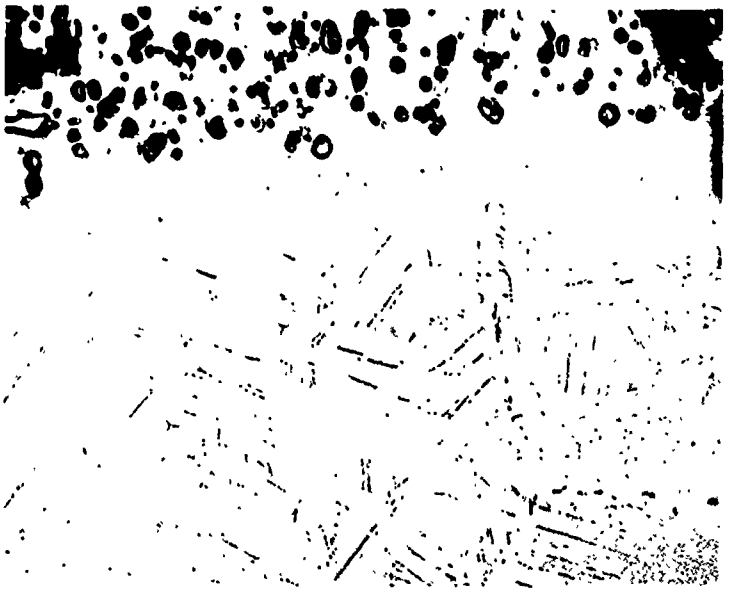


Figure 8

ϵ = 0.4464 mm.

950° C - 140 hours

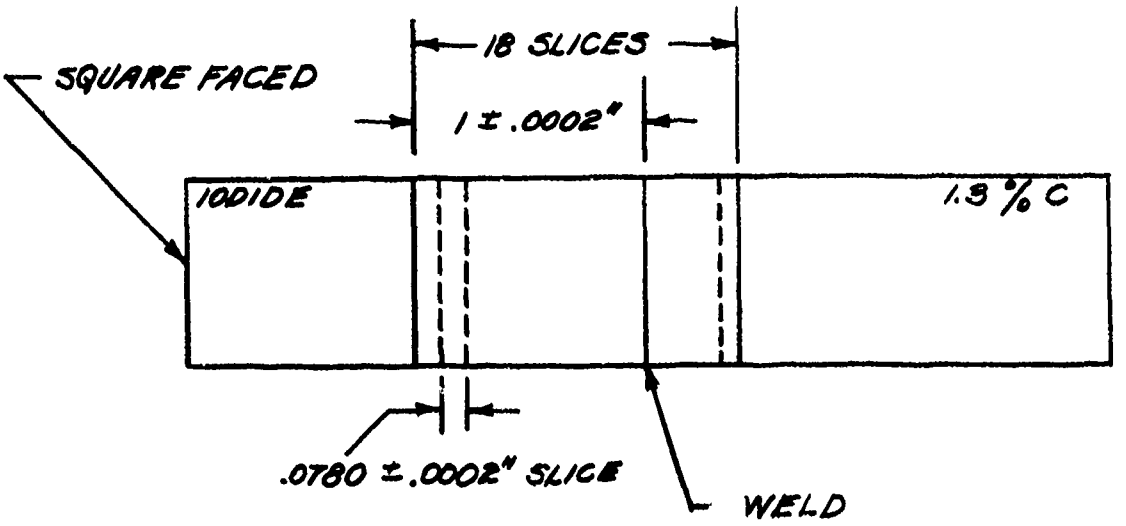
Weld

Figure 9

ϵ = .399 mm.

1050° C - 140 hrs.

Weld



CLEAN MACHINE—CATCH ALL CHIPS IN AL FOIL
SAVE ALL SCRAP

Figure 10. Method of machining layers from annealed diffusion couple for carbon analysis.

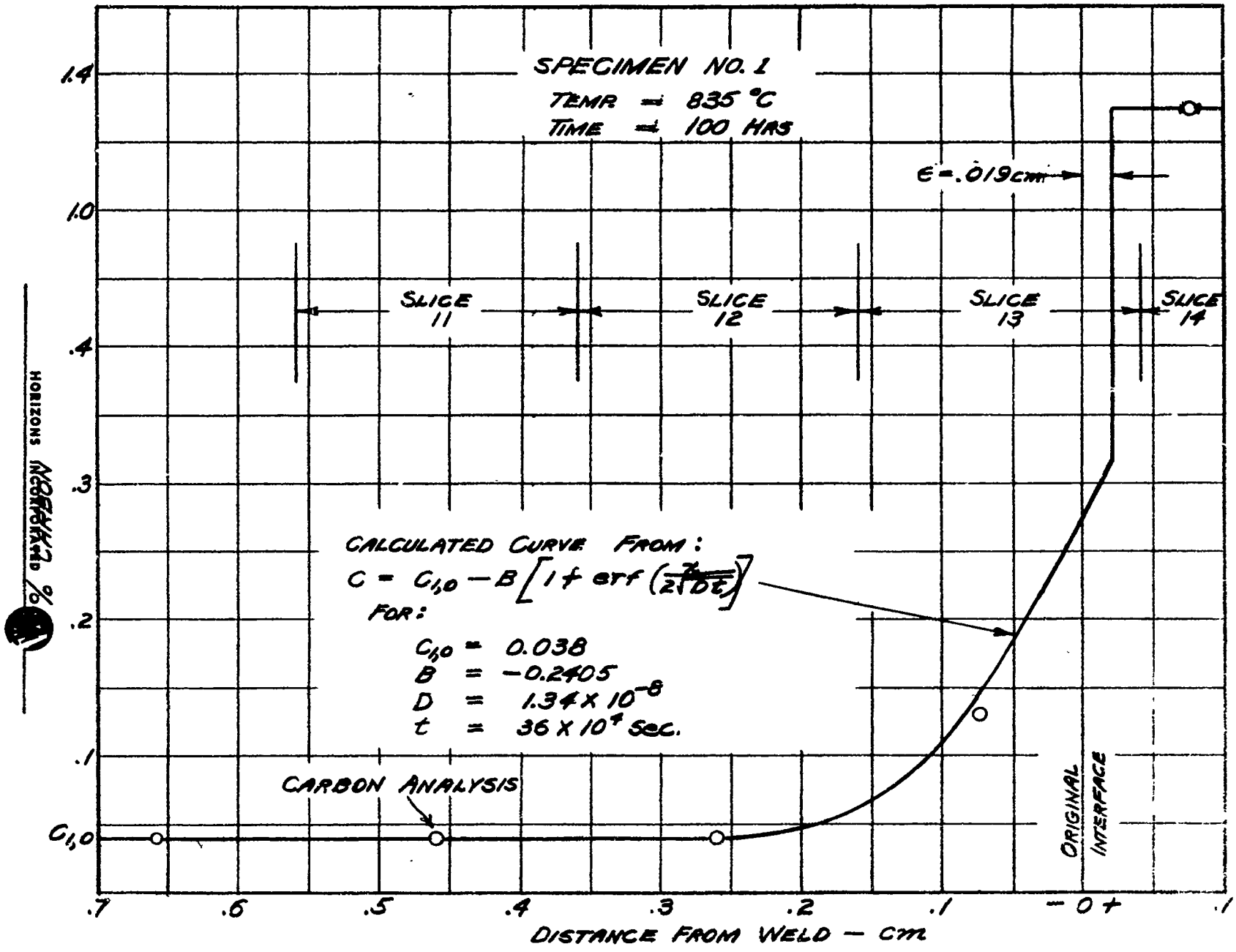


Figure 11. Experimental points plotted for comparison with theoretical curve.

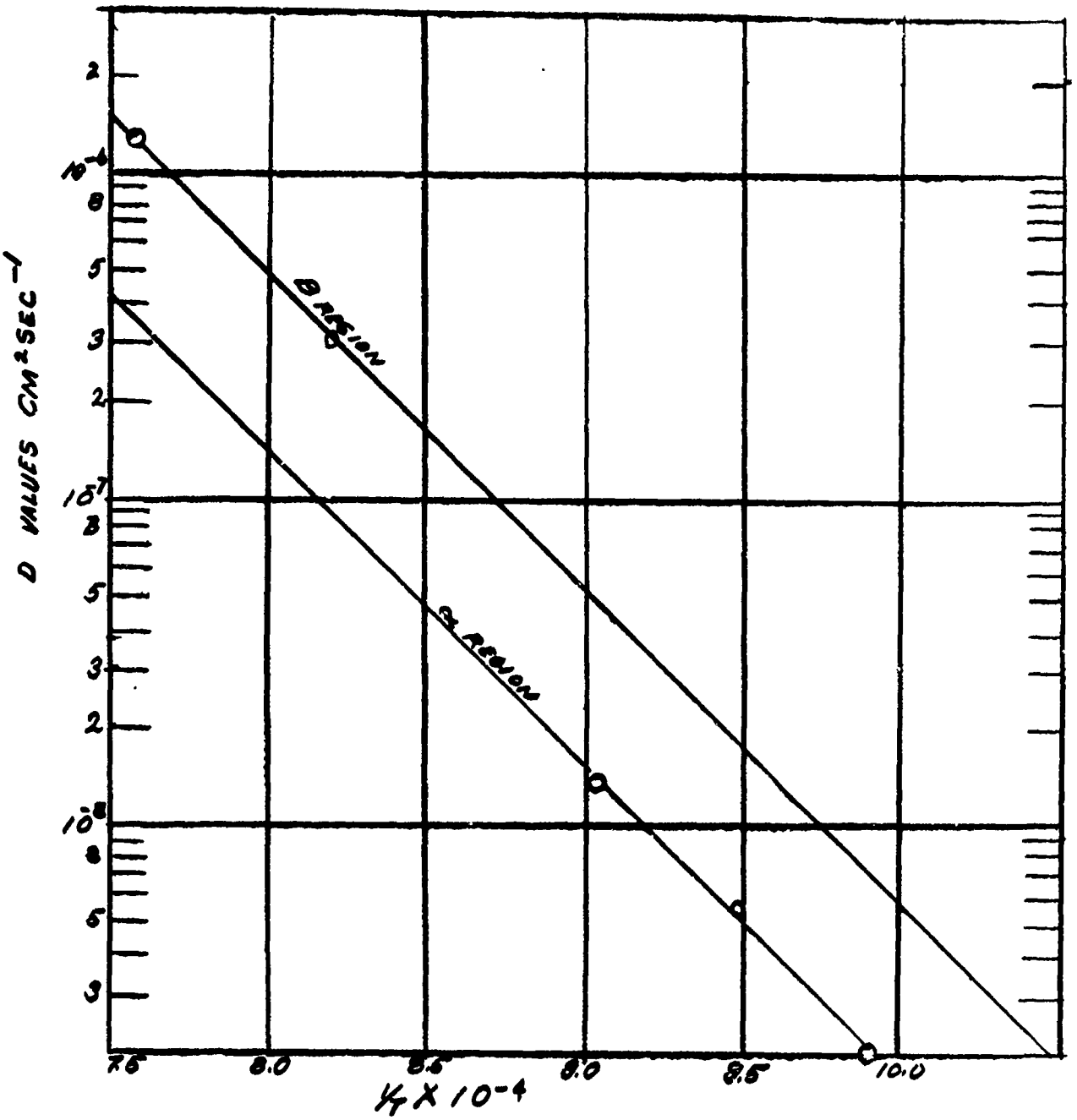


Figure 12. Logarithmic plot of D vs. 1/T